



# Piezoresponse Force Microscopy (PFM) on GaN Nanowires

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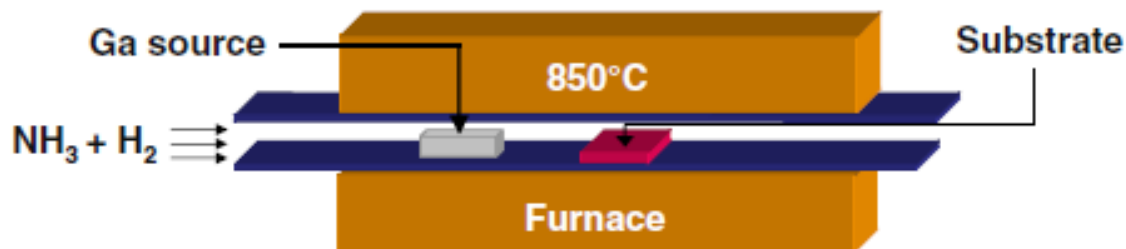


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## Growth of nanowires

- Grown by catalytic CVD using Au or Fe catalyst nanoparticles <sup>1</sup>



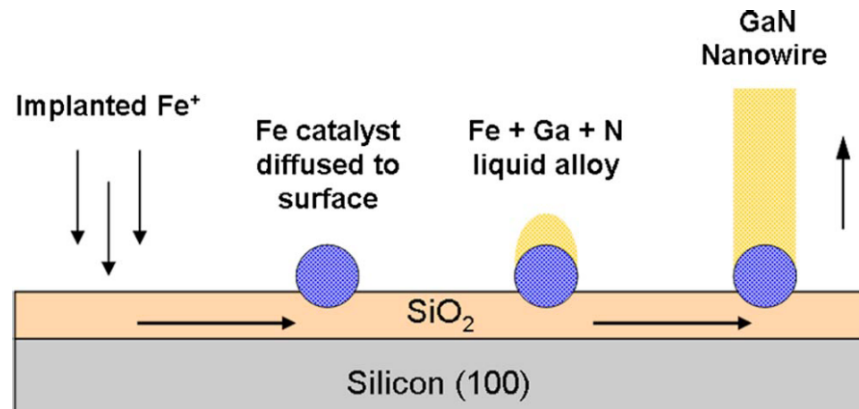
- NH<sub>3</sub> gas flow provides N via :  $2\text{Ga} + 2\text{NH}_3 \rightarrow 2\text{GaN} + 3\text{H}_2$
- Growth time, growth temp, flow rate, flow composition and Ga source/substrate distance influence growth

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## Growth of nanowires

- Growth explained by VLS growth mechanism <sup>2</sup>
  - V: Ga, N from vapor adsorb on nanoparticle surface
  - L: Ga, N and Metal liquid alloy forms around initial particle
  - S : Ga, N attain solubility limit and precipitate solid GaN out of solution

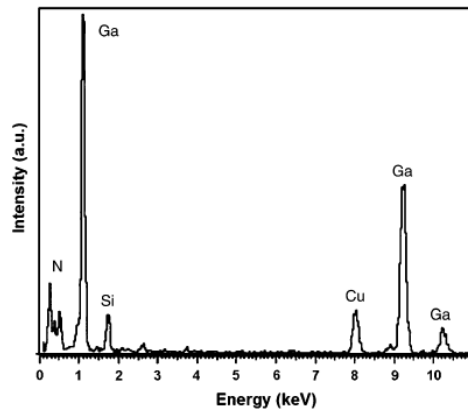


- Single crystal wurtzite nanowires ( $a=0.318\text{nm}$  ,  $c=0.517\text{nm}$ )

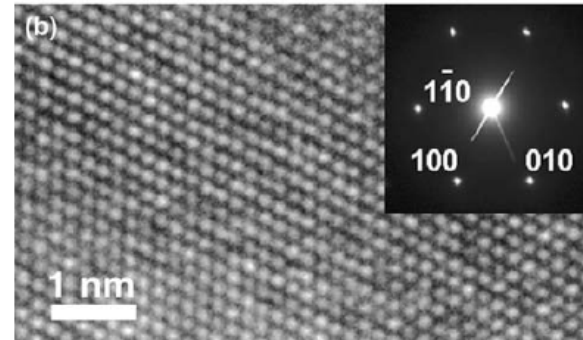
# Piezoresponse Force Microscopy (PFM) on GaN nanowires

## Characterization of nanowires

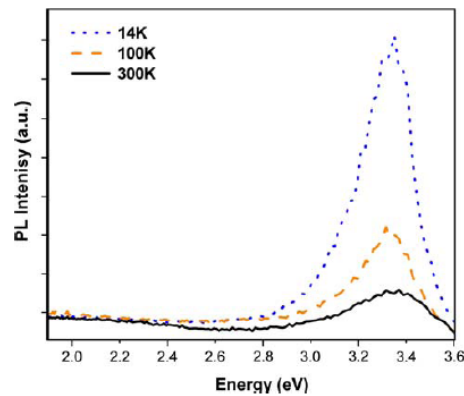
EDS Spectrum <sup>1</sup>:



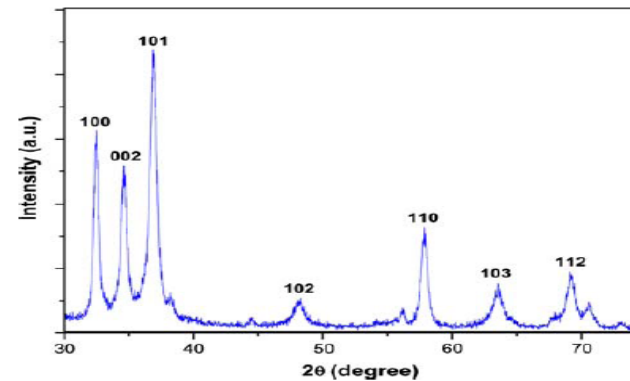
HRTEM <sup>1</sup>:



PL Spectrum <sup>1</sup>:



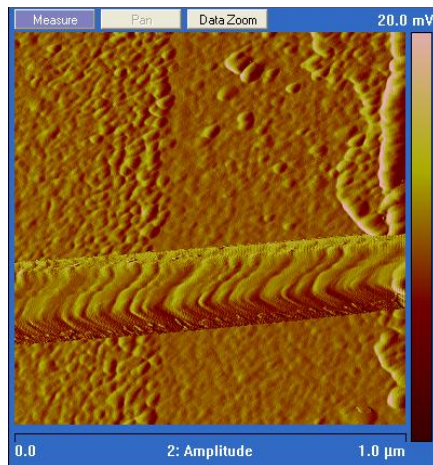
XRD Spectrum <sup>1</sup>:



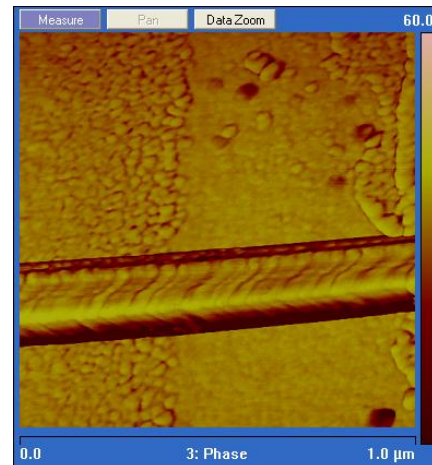
# Piezoresponse Force Microscopy (PFM) on GaN nanowires

## AFM Characterization

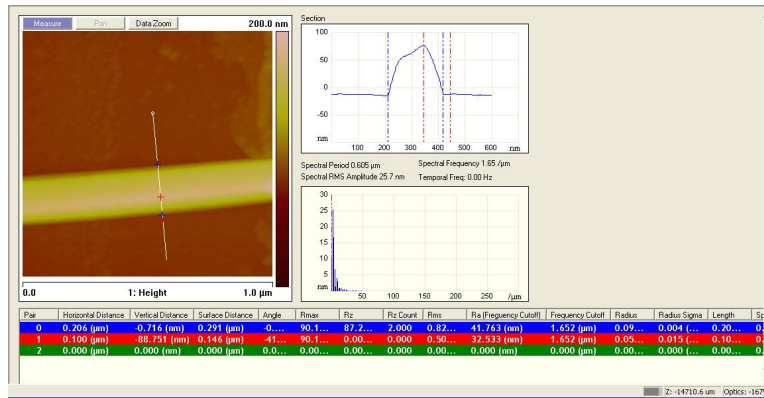
a)



b)



c)



a) Amplitude

b) Phase

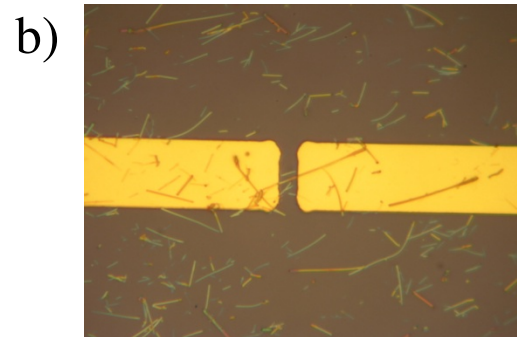
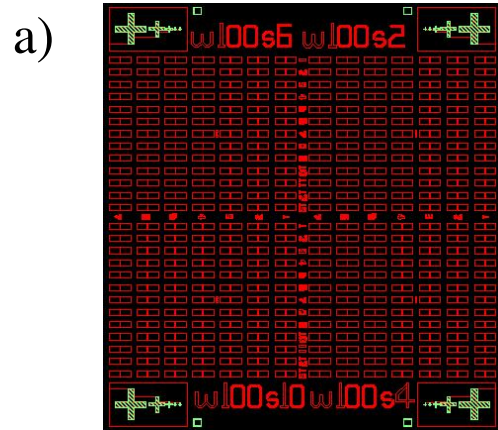
c) Width~200nm,

# Piezoresponse Force Microscopy (PFM) on GaN nanowires

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## Deposition of nanowires on contact grid

- As-grown nanowires dispersed in 2-propanol and sonicated. Suspension deposited on contact grid.



a) Contact grid  
b) Optical bright field image of nanowires

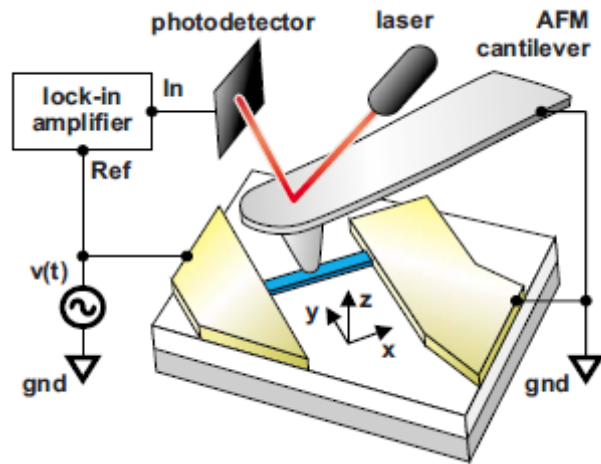
- Ti/Al/Pt/Au contact electrodes patterned by photolithography, e-beam evaporation and subsequent lift-off.



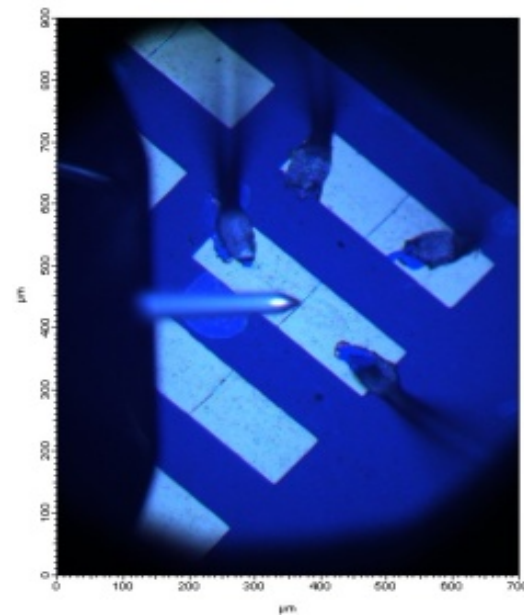
# Piezoresponse Force Microscopy (PFM) on GaN nanowires

## Experimental Set up and Method

- PFM on
  - Unconnected (freestanding) wires
  - Connected wires



a) PFM setup <sup>3</sup>

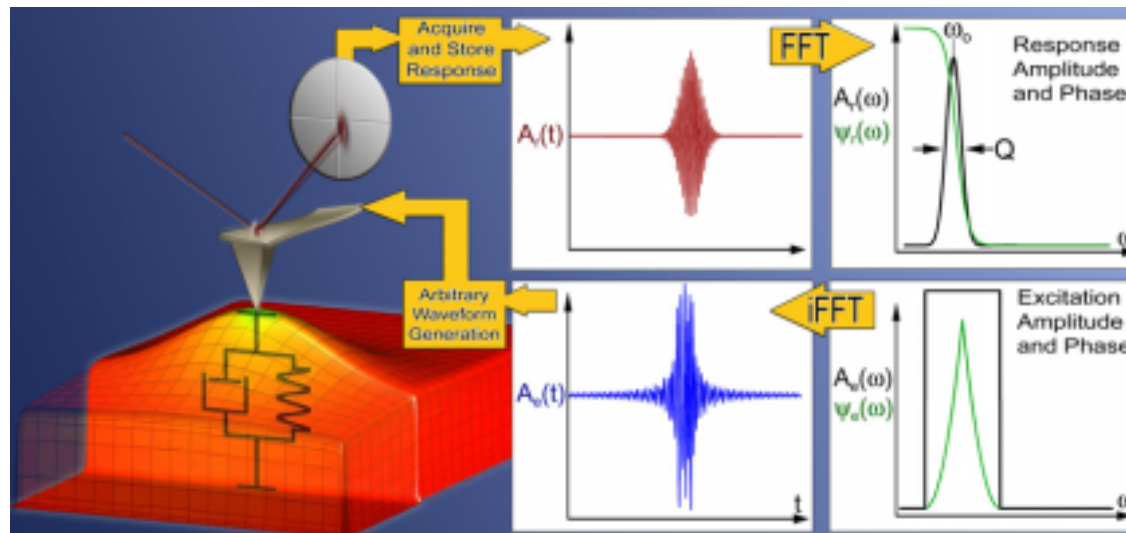


b) Image of wire-bonded contacts and tip used during experiment

# Piezoresponse Force Microscopy (PFM) on GaN nanowires

## Experimental Set up and Method

- Frequency Band Excitation Method<sup>4</sup>
  - Simultaneous excitation and detection within a band of frequencies rather than at a single frequency
  - Only selected regions of Fourier space contain information of interest
  - Method yields full frequency response at each measured point

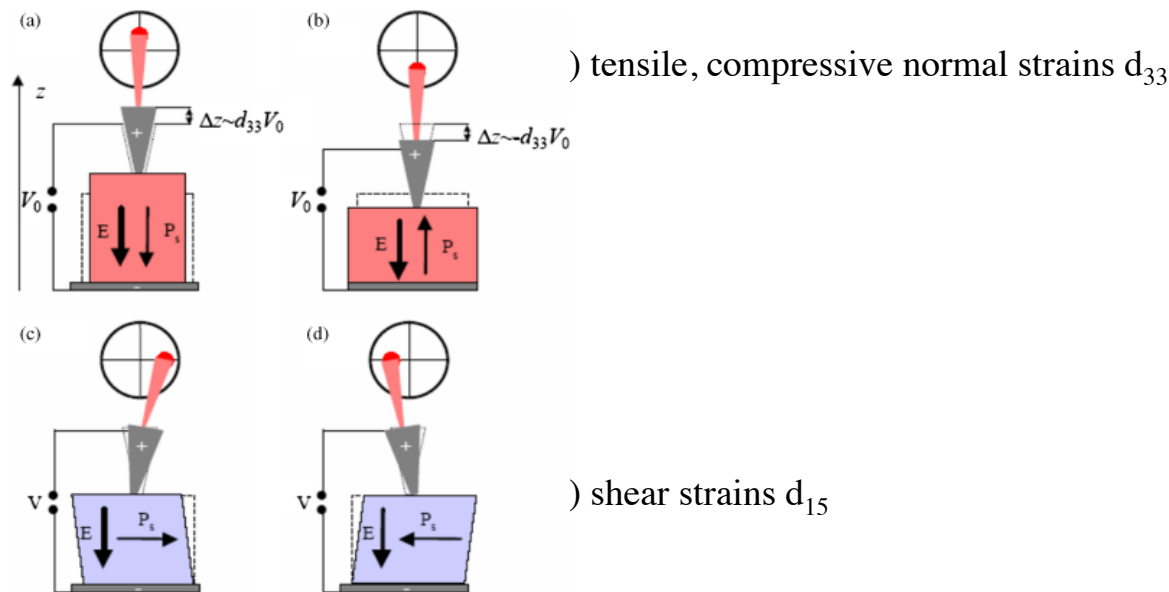




# Piezoresponse Force Microscopy (PFM) on GaN nanowires

## Piezoresponse investigated by PFM (experimental)

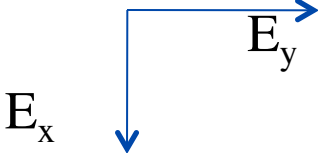
- Relationship (reduced to linear) between electric field,  $E$  and mechanical strain,  $\varepsilon$ :  $\varepsilon_i = d_{ij} E_j$  where  $j=1\dots3$  and  $i=1\dots6$



- Single crystal GaN nanowire polarization along  $[0001]$  direction (figures **c** and **d**)<sup>5</sup>. [Back to slide 10](#)

# Piezoresponse Force Microscopy (PFM) on GaN nanowires

## Piezoresponse investigated using FLOOPS (simulation)

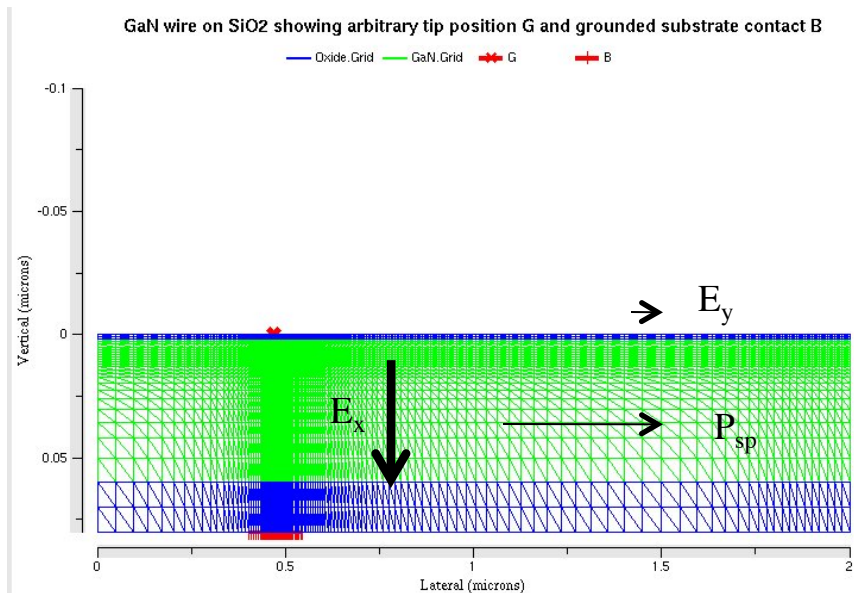
- Assuming axes in 2D as: 
- Nanowire and polarization,  $P_{sp}$  in [0001] parallel to y axis while lying flat. Applied field from tip,  $E_x$  is to  $P_{sp}$  Refer to: [Slide 9 figs.c & d.](#)  
 $E_y$  is to  $P_{sp}$  ||
- Normal deformation comes from the piezoresponse of small volume just underneath the tip
- Assuming constant  $d_{33}$ ,  $d_{13}$ ,  $d_{15}$  in vicinity of tip, resultant measured strain in volume given by :  

$$\epsilon(x) = d_{15} \int E_x(x,y) dx + d_{13} \int E_y(x,y) dy \quad d_{15}=3.1\text{pm/V}, d_{13}=-1.7\text{pm/V}$$

# Piezoresponse Force Microscopy (PFM) on GaN nanowires

## Piezoresponse investigated using FLOOPS (simulated)

- Unconnected GaN nanowire set up:
  - Wire Thickness = 60nm
  - Surface oxide thickness = 2nm
  - SiO2 thickness (which wire is resting on) = 20nm
  - Grounded substrate



Using coefficient matrix:

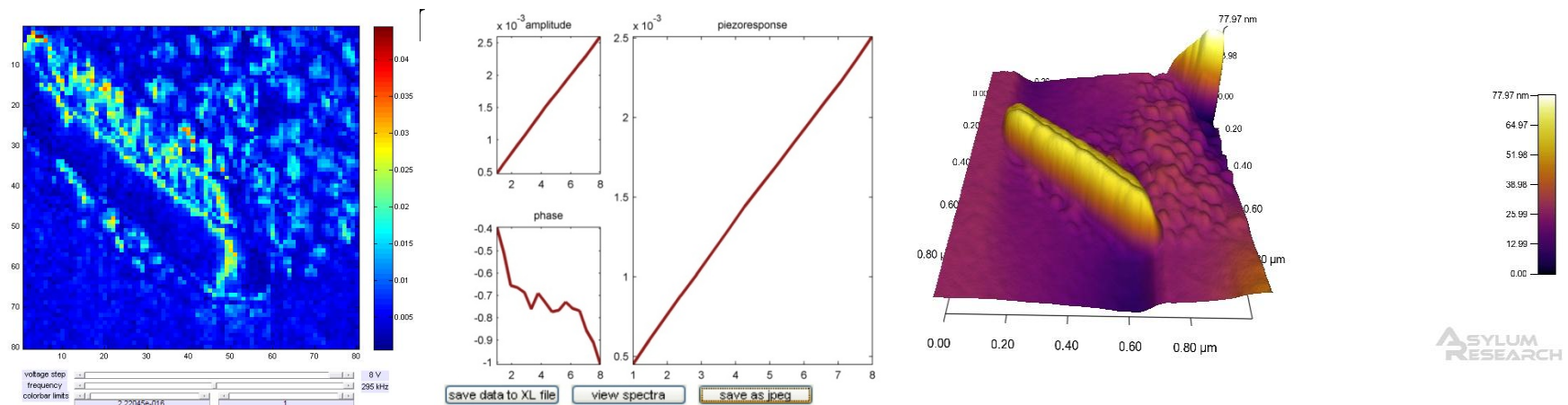
$$\begin{pmatrix} \epsilon_{xx} \\ \epsilon_{yy} \\ \epsilon_{xy} \end{pmatrix} = \begin{pmatrix} 0 & d_{31} \\ 0 & d_{33} \\ d_{15} & 0 \end{pmatrix} \begin{pmatrix} E_x \\ E_y \end{pmatrix}$$

Measured strain =  $\epsilon_{xx} + \epsilon_{xy}$

\* $E_x$  large,  $E_y$  small

# Piezoresponse Force Microscopy (PFM) on GaN nanowires

## Experimental results: Piezoresponse of Unconnected GaN nanowire

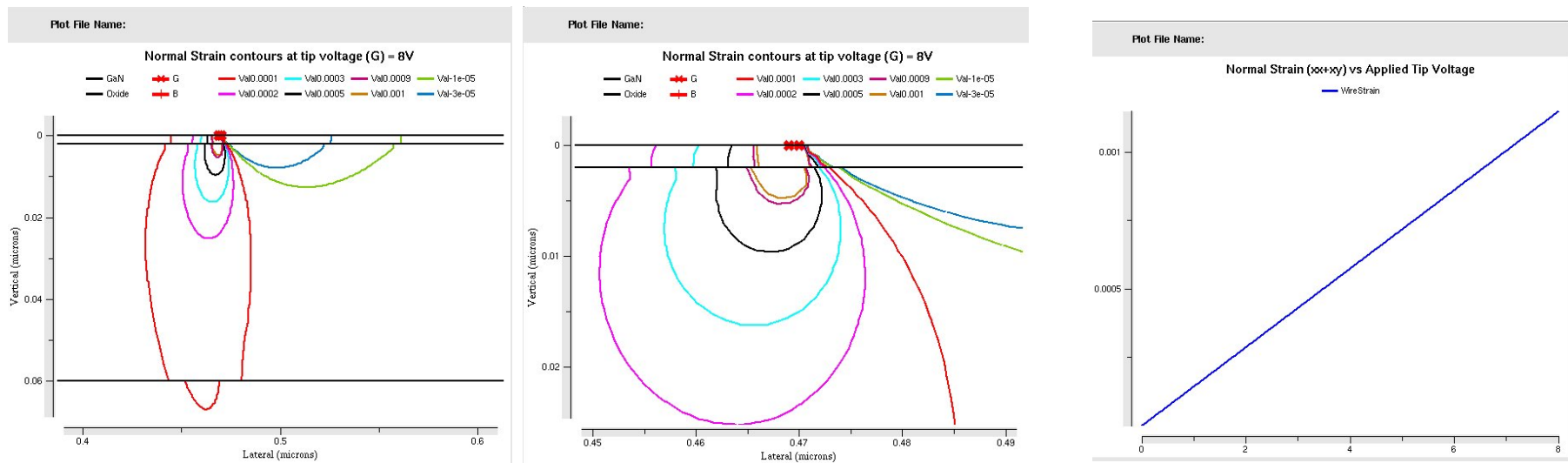


a) Piezoresponse at 8V   b) Hysteresis curves   c) 3D image of wire

- Linear piezoresponse from  $1 \leq V \leq 8$  tip voltage

# Piezoresponse Force Microscopy (PFM) on GaN nanowires

## Simulation results : Piezoresponse of Unconnected GaN nanowire

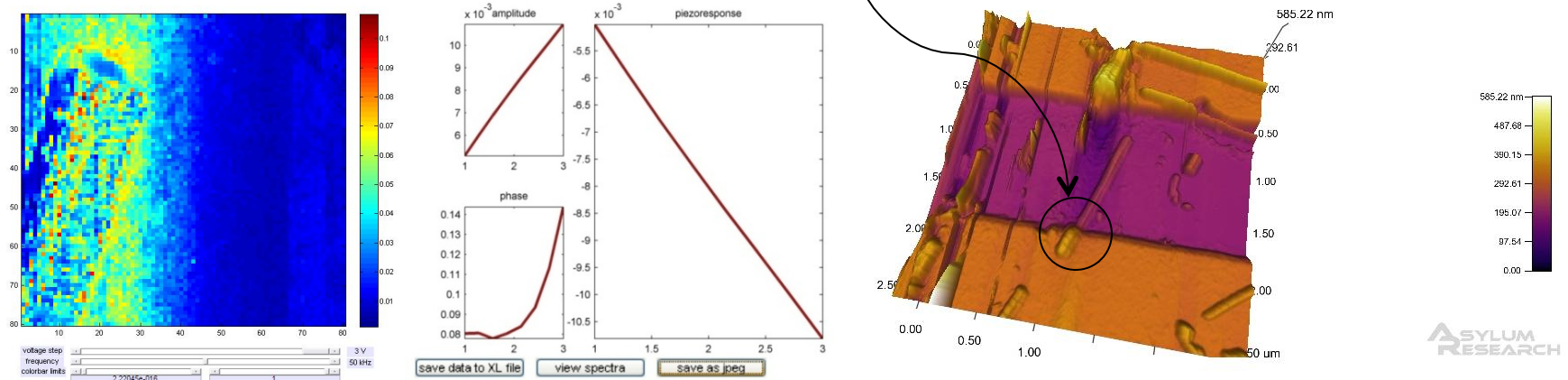


- Wire strain on curve determined by taking an average strain (xx+xy)
  - \*of 7 points in a 4nm x 4nm area directly underneath tip contact
- \* No clamping effect for free standing wire,

# Piezoresponse Force Microscopy (PFM) on GaN nanowires

## Experimental results: Piezoresponse of Connected GaN nanowire

Connected wire (1 side grounded)



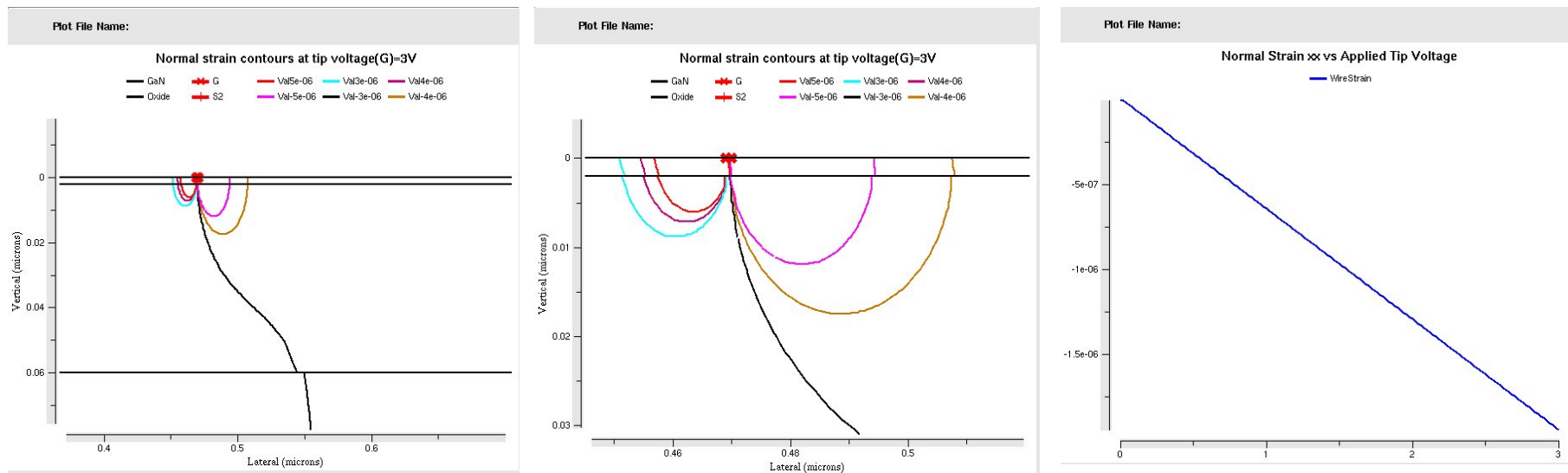
a) Piezoresponse at 3V   b) Hysteresis curves   c) 3D image of wire

- Linear piezoresponse from  $1 \leq V \leq 3$  tip voltage



# Piezoresponse Force Microscopy (PFM) on GaN nanowires

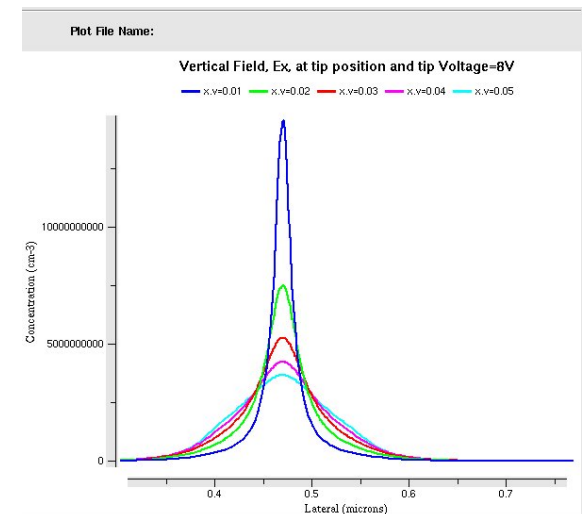
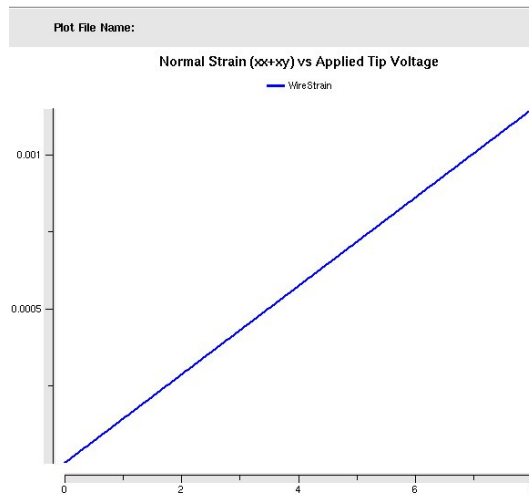
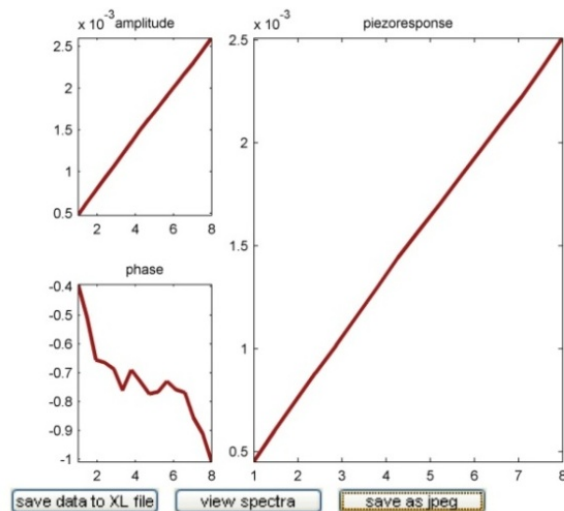
## Simulation results : Piezoresponse of Connected GaN nanowire



- Wire strain on curve determined by taking an average strain (xx)\* of 7 points in a 4nm x 4nm area directly underneath tip contact
- \* Contact restricts shearing forces, so xy component is removed due to clamping effect

# Piezoresponse Force Microscopy (PFM) on GaN nanowires

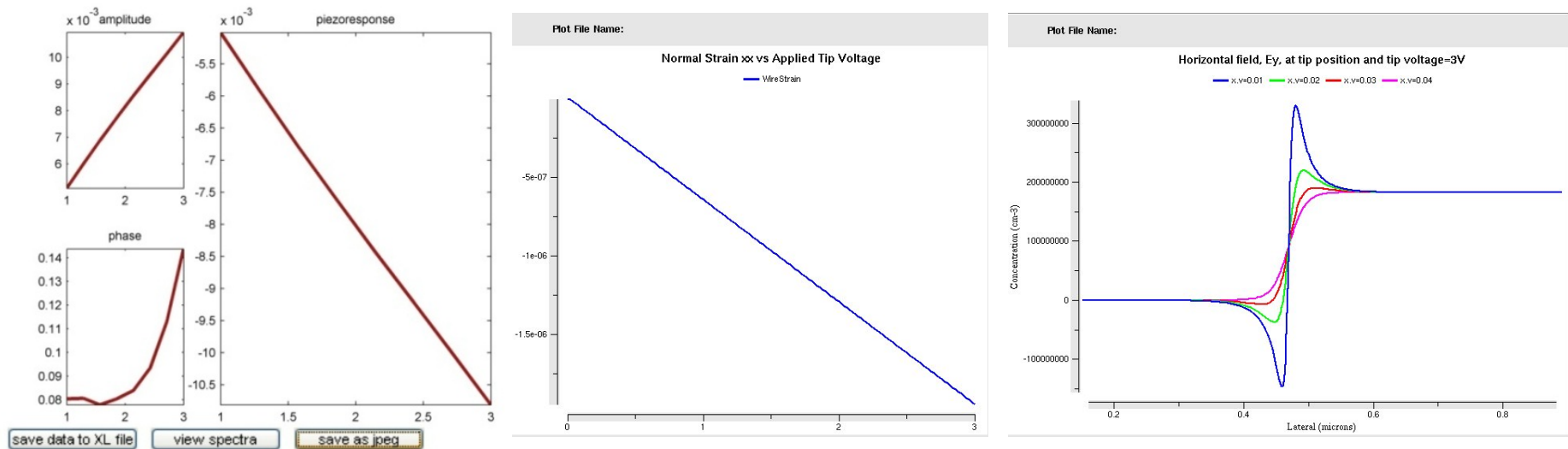
## Experimental vs Simulated comparison for Unconnected GaN nanowire



- Experimental and Simulated piezoresponse at 8V are  $2.5 \times 10^{-3}$  and  $1.14 \times 10^{-3}$  respectively. Decent agreement between the two.
- For unconnected wire,  $E_x$  dominates,  $E_y$  is negligible so **Shear** strain,  $d_{15}$  dominates the measured response.

# Piezoresponse Force Microscopy (PFM) on GaN nanowires

## Experimental vs Simulated comparison for Connected (1 side) GaN nanowire



- Experimental and simulated piezoresponses show same trend. Clamping (displacement=0 at edge) causes shear strain contribution from  $E_x$  to be minimized.
- For connected wire,  $E_y$  determines strain. Coeff.  $d_{13}$  (Poisson's ratio) determines the measured response.

# Piezoresponse Force Microscopy (PFM) on GaN nanowires

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## Conclusions and Further Work

- Initial 2D strain simulations using coefficients listed for bulk GaN in reasonable agreement with nanowire piezoresponse measured by experiments.
- 3D simulation needed for more accurate comparison.
- Measurement on fully connected (2 sides) GaN nanowires
- Simulation of experimentally measured hysteresis effect.
- Extraction of accurate coefficients specific to GaN nanowires difficult due field superposition and inevitable clamping effect

# Piezoresponse Force Microscopy (PFM) on GaN nanowires

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## References

- 1] Jason L. Johnson, Yongho Choi, Ant Ural , Wantae Lim, J.S. Wright, B.P. Gila, F. Ren and S.J. Pearton: *Growth and Characterization of GaN Nanowires for Hydrogen Sensors* ,J. of Elec. Materials. Vol 38, No. 4, 2008
- 2] Jason L. Johnson, Yongho Choi, and Ant Ural: *GaN nanowire and Ga<sub>2</sub>O<sub>3</sub> nanowire and nanoribbon growth from ion implanted iron catalyst* J. Vac. Sci. Technol. B Vol 26, No.6, 2008
- 3] J. Wang, C. Stampfer, C. Roman, W. H. Ma, N. Setter, C. Hierold: *Piezoresponse force microscopy on doubly clamped KNbO<sub>3</sub> nanowires* , Applied Phys. Lett. Vol 93, No 223101, 2008
- 4] Stephen Jesse, Sergei V Kalinin, Roger Proksch, A P Baddorf, B J Rodriguez: *The band excitation method in scanning probe microscopy for rapid mapping of energy dissipation on the nanoscale*, Nanotechnology Vol 18, 2007
- 5] Nina Balke, Igor Bdikin, Sergei V. Kalinin, Andrei L. Kholkin: *Electromechanical Imaging and Spectroscopy of Ferroelectric and Piezoelectric Materials: State of the Art and Prospects for the Future*, J. Am. Ceram. Soc., Vol 92, No.8, 2009

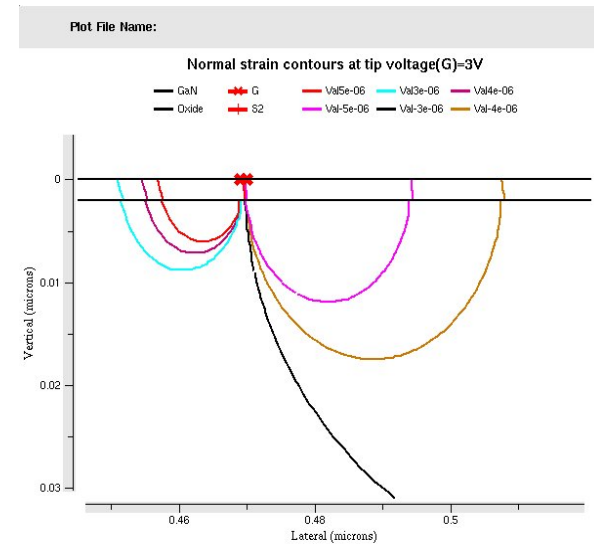
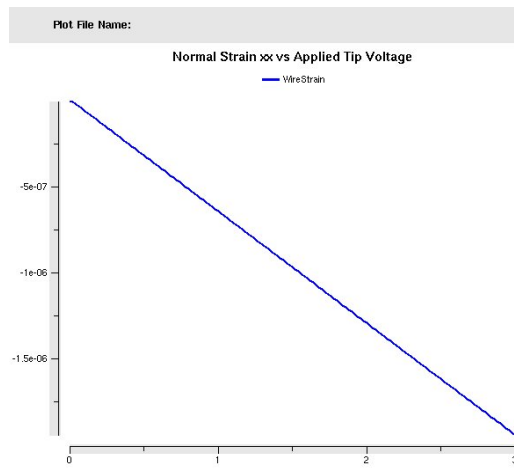
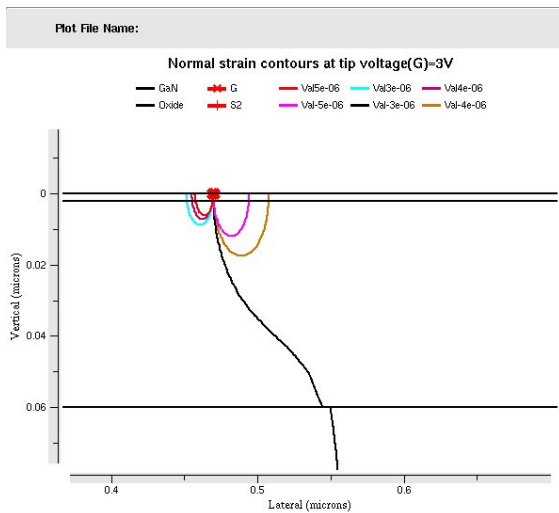
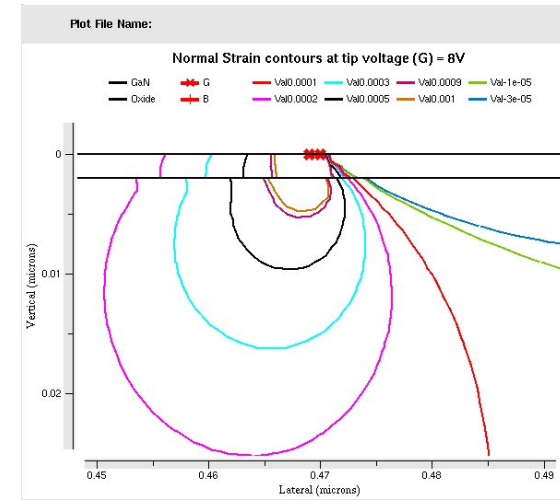
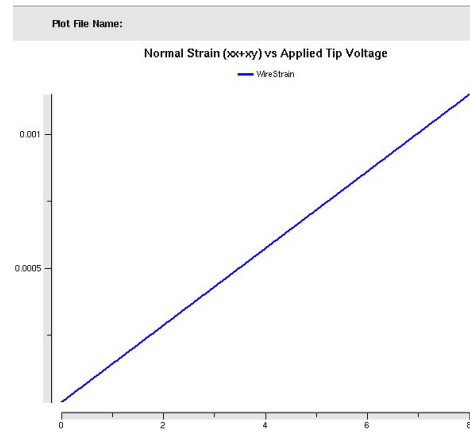
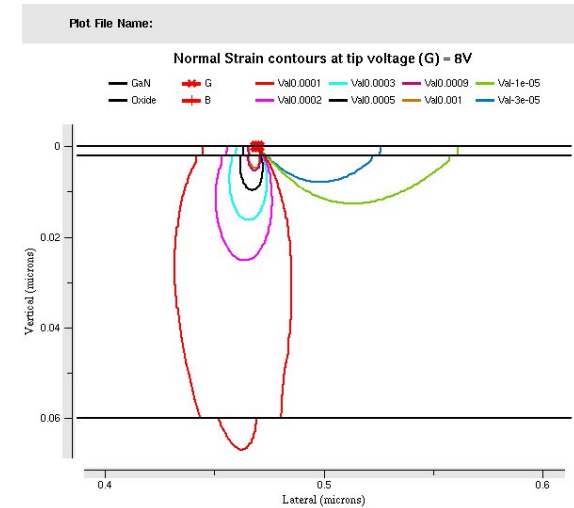
# Back-up Slides

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- Slide 21 - Simulation pictures
- Slide 22 – Experiment results showing hysteresis



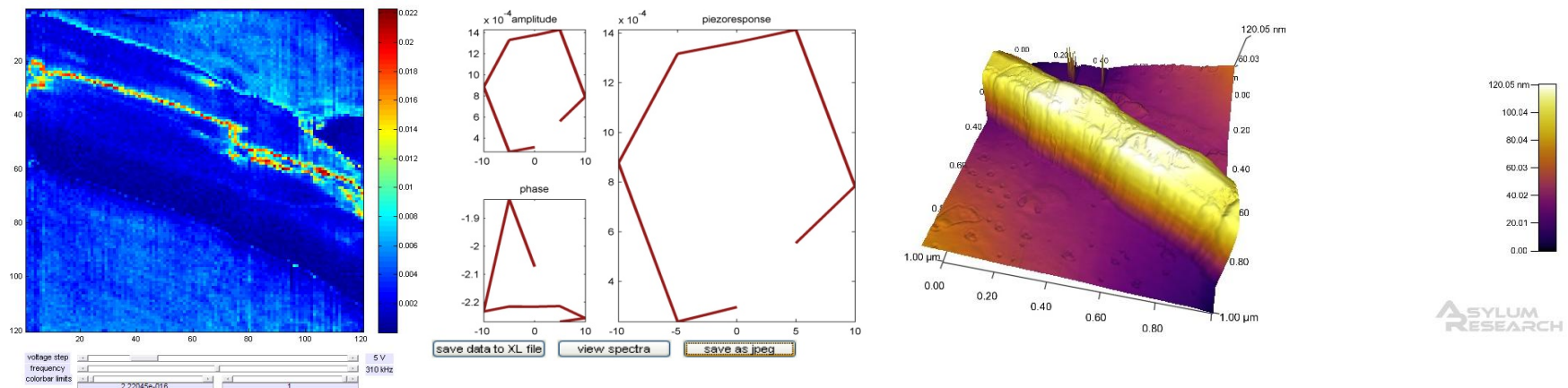
# Unconnected and connected wire simulation pictures



# Piezoresponse Force Microscopy (PFM) on GaN nanowires

## Experimental results: Piezoresponse with Hysteresis

- Unconnected GaN nanowire



- a) Piezoresponse at 5V      b) Hysteresis curves      c) 3D image of wire

- Hysteresis from  $+5 \leq V \leq 0$  tip voltage swing